Arsenic (V) removal from aqueous system using adsorbent developed from a high iron-containing fly ash

Yi Li, Fu-Shen Zhang^{*}, Fu-Rong Xiu

Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, 18 Shuangqing Road, Beijing 100085, China

ARTICLE INFO

Article history:

Received 16 February 2009

Received in revised form 6 July 2009

Accepted 10 July 2009

Available online 3 August 2009

Keywords:

Arsenate

Fly ash

Loading mechanism

Ferric oxide

ABSTRACT

A novel adsorbent for arsenic (V) removal from wastewater was developed through simple chemical processes using a special iron-abundant fly ash. In the synthesis process, the inherent iron in the fly ash was rearranged and loaded on the surface of the fly ash by dissolution and precipitation processes. The adsorbent (HIOFAA) was characterized by XRD, FT-IR, SEM, LPS and BET surface area. The results showed that porous amorphous FeOOH was loaded on the surface of HIOFAA successfully. The BET surface area of HIOFAA was 22 times of those of the original fly ash, and furthermore, the mean particle size of HIOFAA increased 3 times compared to the raw fly ash, thus effectively accelerated the solid/liquid separation after the adsorptive treatment. The adsorption isotherm data could be well described by Langmuir isotherm model, and the adsorption capacity for arsenic removal was 19.46 mg g^{-1} . Accordingly, it is believed that the adsorbent developed in this study is effective for arsenic polluted wastewater treatment.

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1. Introduction

Arsenism has become one of the most serious endemic diseases in China in the last decade due to arsenic pollution. It was reported that over 2 million people are exposing to arsenic polluted water and about 10,000 persons were diagnosed as arsenism patients (Sun, 2004; Kanel et al., 2005). Elevated arsenic concentrations in groundwater are mainly caused by human activities and natural processes, e.g., mining, release of industrial wastewater and disposal of chemical wastes. In recent years, economic development in China has led to the annual increase of quantity of discharged wastewater containing high arsenic concentration. Currently, a variety of treatment methods have been intensively researched for arsenic removal from contaminated water. The major methods involve adsorption, ion exchange, nanofiltration, reverse osmosis, electrodialysis, precipitation–coagulation, oxidation–precipitation, and biological treatment (Zhang and Itoh, 2005; Baciocchi et al., 2005; Han et al., 2002; Kang et al., 2000; Weng et al., 2005; Meng et al., 2002; Zhang and Itoh, 2006; Katsoyiannis et al., 2002). Among them, adsorption using a variety of iron-containing materials has been proven to be economical, effective and easy for operation. Thus several iron-containing materials, such as amorphous hydrous iron oxide (FeOOH), goethite (α -FeOOH) and akaganeite (β -FeOOH) (Raven et al., 1998; Lakshminathiraj et al., 2006; Solozhenkin et al., 2003), have been examined and proven to be promising adsorptive materials due to their removing efficiency.

Among these materials, amorphous hydrous iron oxide has the highest adsorption capability since it has the highest surface area. However, this iron-containing adsorbent is merely available as fine powders or exists as gel or suspension in aqueous solution, hence it is difficult to separate entirely from the treated solution after the adsorption reactivity. In order to overcome this shortage, some matrices were selected to support or load those iron oxides. The matrices include municipal solid waste (MSW), cement, sand, activated alumina (AA), silica oxide and alginate beads (Zhang and Itoh, 2005; Kundu and Gupta, 2002; Hsua et al., 2008; Guo et al., 2007; Vaishya and Gupta, 2002; Kuriakose et al., 2004; Zeng, 2003; Zouboulis and Katsoyiannis, 2002). Previous studies showed that these synthetic Fe-loaded adsorbents had good affinities and high removal efficiency for arsenic.

In a previous study, we successfully loaded amorphous FeOOH on the surface of municipal solid waste melted slag through chemical reactions and high temperature coating treatments (Zhang and Itoh, 2005). Moreover, Fan et al. (2008) have synthesized another iron-impregnated adsorbent for arsenic removal using the MSW and coal co-combustion ash. Among the foregoing studies, chemical agents, such as FeCl_3 , FeSO_4 , were necessary for the adsorbents synthesis. Thus high iron-containing waste materials are desirable for the production of the adsorbents since they can avoid adding the iron source in the process of synthesis. Recently, we have found a new type of coal fly ash with iron oxide as high as 17.52%. This type of ash is an ideal Fe-containing raw material for the synthesis of adsorbent for arsenic removal.

The aim of this study was to develop a new route for effective utilization of the special type of fly ash. A novel adsorbent, containing high amount of amorphous FeOOH on the surface of fly ash, was

^{*} Corresponding author. Tel./fax: +86 10 62849515.

E-mail address: fszhang@rcees.ac.cn (F.-S. Zhang).

synthesized using simple chemical reactions. The adsorbent was applied for arsenic removal from aqueous system, and the optimum conditions were established.

2. Experimental

2.1. Materials

Fly ash was sampled from Gaobeidian thermal power plant (4×200 MW) located in Beijing (China). The ash was verified to be safe for the environment and has realized the commercial utilization in the traditional areas such as concrete, pavement and dam. The ash was collected by an electrostatic precipitator after the pulverized coal burnt in the cyclone furnace boiler. After sampling, part of the ash was dried at 105 °C for 24 h and stored in a desiccator for use. Typical chemical composition of the ash is listed in Table 1.

Stock solution containing 1000 mg l⁻¹ of arsenic (V) was prepared from analytical pure Na₃AsO₄·12H₂O following the procedure: Firstly, Na₃AsO₄·12H₂O was directly dissolved into a 10 ml of distilled water, then transferred to a 1-l volumetric flask and diluted to a pre-determined volume. Arsenic working solutions were freshly made by diluting stock solution with distilled water. HCl (1 M, 0.1 M), HNO₃ (1 M, 0.1 M) and NaOH (1 M, 0.1 M) solutions were prepared for pH adjustment.

2.2. Adsorbent synthesis

According to Fan et al. (2005), some alkali and alkaline earth elements, such as K₂O, Na₂O, CaO and MgO, were removed from the raw ash by washing the sample with hot water so as to reduce the consumption of hydrochloric acid in the process of adsorbent synthesis.

The high iron oxide fly ash adsorbent (HIOFAA) was prepared according to the following procedure: Firstly, 3.0 g of raw fly ash was added to 250 ml of water, and the mixture was stirred and heated to 95 °C for 1 h, then the insoluble part in the mixture was filtrated and rinsed repeatedly until the pH of leachate remained 6.0–7.0. The insoluble material obtained was dried at 100 °C for 2 h. Secondly, the pretreated fly ash was contacted with 100 ml of 1 M HCl solution, vibrated for 2 h and placed steadily at 60 °C for 0.5 h. The color of the solution gradually changed to yellow from transparency. Thirdly, 1 M NaOH solution was added dropwise into the solution until the final pH was adjusted to about 6.0, and the orange gel appeared in the solution. The mixture continuously was vibrated for 1 h, and then aged at 75 °C for 3 days. Finally, the slurry was dewatered by centrifugation and continuously rinsed for several times with distilled water (100 ml of water was used per washing cycle) until the pH of the leachate remained constant in three consecutive rinses, then dried at 75 °C for 24 h. Upon cooling, 3.43 g of composite solid was obtained. The solid was broken to separate the powders in the carnelian mortar, and afterwards stored in the capped polyethylene bottle for use.

2.3. Analysis

In order to discuss the loading mechanisms of HIOFAA, raw fly ash (RFA), acidized fly ash (AFA) and the final product HIOFAA were examined, among which, AFA was obtained by the following procedure: after the raw fly ash was acidized adequately at 60 °C for 0.5 h by HCl solution (above mentioned), the remnant insoluble part was separated from the mixture by centrifugation, then washed for

three times with distilled water, and finally dried at 75 °C for 24 h. The final solid power obtained was identified as acidized fly ash (AFA).

The X-ray diffraction (XRD) patterns of RFA, AFA and HIOFAA were obtained using X-ray diffractometer (Philips PW 1700, Holland). The operating conditions were 45 kV and 250 mA, using Cu Kα radioactive source. The samples were scanned in the range of 10° to 60°, and JCPDS (Joint Committee on Powder Diffraction Standards) files were used for the identification of crystalline compounds. The surface morphology of RFA, AFA and HIOFAA were obtained using scanning electron micrograph (SEM) (Hitachi S-3000 N, Japan). The functional groups of RFA, AFA and HIOFAA were tested by the technology of Fourier transform infrared spectrometry (FT-IR) (Nicolet Nexus670, USA) in the wavenumbers of 4000–400 cm⁻¹. The technology of laser particle size (LPS) was used to analyze the particle size distribution of the three samples by the laser particle sizer (Master size 2000, Malvern Co, UK). The BET (Brunauer-Emmet-Teller) specific surface area was determined by fitting the linear portion of the BET plot to BET equation, and pore size distribution was calculated based on the desorption plot of N₂ adsorption–desorption isotherm using the Barrett-Joyner-Halenda (BJH) method (Micromeritics ASAP 2000, USA). Skeleton density and porosity of HIOFAA and AFA were measured based on the water displacement method proposed in Black. Meanwhile, porosity was calculated using the equation (Lin and Wu, 2001)

$$\varepsilon_p = 1 - \frac{\rho_s}{\rho_p} \quad (1)$$

where ε_p is the porosity of adsorbent, ρ_s is the skeleton density of adsorbent (g cm⁻³) and ρ_p is the density of AFA (g cm⁻³).

After the adsorptive reaction, the supernatants were collected and arsenic concentration was examined by inductively coupled plasma optical emission spectrometer (ICP-OES) (PerkinElmer OPTIMA 2000, USA).

2.4. Batch experiment

In order to optimize arsenic removal conditions, the effects of aging time, solution pH, arsenic initial concentration, contact time and HIOFAA dosage on arsenic removal were comprehensively tested. Specifically, a series of 50-ml capped polyethylene bottle were shaken in a shaker at room temperature (nearly 25 °C) and 220 rpm. In each test, 25 ml of arsenic solution was loaded in the bottle. Arsenic concentrations were in the range of 1.0 to 100 mg l⁻¹ and HIOFAA dosage was in range of 2.0 to 60 g l⁻¹. The pH (from 1.5 to 13) value was adjusted to the desired level with HNO₃ or NaOH solution. During the adsorption process, the test bottles were shaken for a fixed time (from 2.0 to 30 h), then the suspensions were immediately centrifuged to separate the solid from the solution. The supernatant were sampled and then analyzed by ICP-MS for the equilibrium arsenic concentration. Arsenic removal was calculated by the difference between the concentrations of arsenic before and after adsorption. Arsenic concentrations in unit of mg l⁻¹, are all given as elemental arsenic.

3. Results and discussion

3.1. Characterization of the adsorbents

The X-ray diffraction (XRD) patterns of RFA, AFA, and HIOFAA are showed in Fig. 1. The main diffraction peaks of RFA are assigned to quartz (SiO₂), hematite (Fe₂O₃), Anhydrite (CaSO₄), Magnetite (Fe₃O₄) and lime (CaO). However, different from previous report (Jing et al., 2008), aluminium mineralogical phrases, such as mullite, could not be found although the composition of Al₂O₃ in the raw fly ash is as high as 25.70% (Table 1). In the XRD pattern of AFA, the quartz

Table 1
Chemical composition of the fly ash employed in this study.

Properties	LOI	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O
Percentage (Mass, %)	0.27	29.33	25.70	17.52	22.17	1.60	4.15	0.56	1.81

Fig. 1. X-ray diffraction patterns of RFA, AFA and HIOFAA.

becomes the main crystalline phase and minor hematite, magnetite and Anhydrite is still existed. This shows that the content of quartz increases and the mineral containing elemental Fe (hematite and magnetite) decreases after the process of acidic treatment. In the XRD pattern of HIOFAA, no new diffraction peaks appear and no new crystalline phase is observed in contrast with AFA, and moreover, the diffraction peaks of magnetite disappear, indicating that the loaded new substance in the surface of the HIOFAA is amorphous. Meanwhile, the magnetite on the surface of AFA is covered up and hereby XRD diffraction peaks vanish entirely.

Fig. 2 illustrates the FT-IR transmittance spectra of RFA, AFA and HIOFAA. In contrast with RFA, the adsorption at 1103 cm^{-1} assigned to the vibration of Fe–O bond (Fan et al., 2008), and the band at 997 cm^{-1} assigned to the asymmetric stretching vibration of T–O (T = Si, Al) bonds (El-Naggar et al., 2008), faint dramatically, showing that a great portion of iron oxide and aluminium oxide dissolved into the HCl solution, but the typical bands (1084 , 785 and 461 cm^{-1}) of quartz (Fernández-Jiménez and Palomo, 2005), become more obvious, which could attribute to the inertness of quartz and thereby

enrichment in the AFA. In the spectra of HIOFAA, the new bands at 3486 and 1662 cm^{-1} is assigned to O–H vibration, the former corresponds to O–H stretching vibration and the latter to deformation vibration bond (Elizalde-González et al., 2001), implying that the new substance loaded in the HIOFAA exists in the form of hydroxy compound.

The morphology changes of RFA, AFA and HIOFAA are showed in Fig. 3. The images of HIOFAA show that the final powder is drastically different from the original ash. The surface of HIOFAA is coated entirely by the hydroxy compounds and gets into porous network structure, while the size of granule also increases sharply. LPS analysis shows that the mean particle size of RFA and HIOFAA are 5.51 and $21.05\text{ }\mu\text{m}$, respectively. The final product is nearly 4 times as large as RFA, which can efficiently accelerate separation effect after adsorptive treatment. In a blank test for comparison, equivalent RFA replaced HIOFAA to remove arsenic from the solution in the same conditions. After the treatment and vibration, the solution was turbid and it took more than 2 h for the solution to become clear, but it took only several minutes for HIOFAA to get clear.

Fig. 2. FT-IR spectra of RFA, AFA and HIOFAA.

Fig. 3. SEM images of RFA, AFA and HIOFAA.

The nitrogen adsorption-desorption isotherm of HIOFAA with the pore size distribution is shown in Fig. 4. The Type IV adsorption isotherm of HIOFAA and its hysteresis loop imply the mesoporous structure of material. The pore size distribution confirms that the pore size of HIOFAA ranged from 21 Å to 505 Å, the average pore size was 23 Å, and porosity was 17.8%.

3.2. Loading mechanisms

Fly ash is rich in aluminum and iron oxides, and is thought to be very important source of pre-mined material, hence many researchers have studied the ash leachability under the acidic conditions for the extraction of alumina, iron, and the other heavy metal elements (Matjie et al., 2005; Baba and Kaya, 2004; Sočo and Kalemekiewicz, 2007). When fly ash contacts with hydrochloric acid, most of the metals, such as Fe and Al, can leach into the solution through the following reactions:

During the reaction, the color of mixture in the conical flask gradually changed to light yellow due to the dissolution of Fe^{3+} in the fly ash. When the slurry was adjusted with sodium hydroxide solution to raise its alkalinity, Fe^{2+} , Fe^{3+} and Al^{3+} precipitated through the

following reactions, the slurry of mixture became orange from light yellow, indicating that Fe ion was precipitated from the solution.

Literature shows that Fe and Al ions precipitate from aqueous solution when pH value of solution is adjusted to 3.0 and 3.5, respectively (Matjie et al., 2005). In our experiments, the precipitation of white $\text{Fe}(\text{OH})_2$ was firstly observed in the solution, but it changed into orange simultaneously, indicating that $\text{Fe}(\text{OH})_2$ was oxidized to $\text{Fe}(\text{OH})_3$.

When fly ash contacts with acidic solution, the bonds of Si–O on the surface of spherical glassy particle increase due to dissolution of the other metal elements. Moreover, when FeOOH precipitate from the aqueous solution and coated on the surface of residual fly ash, Fe–Si surface complex (typically, $\text{Fe}-\text{O}-\text{Si}(\text{OH})_3$) occur between Fe and Si in situ as reported in the previous literature (Swedlund and Webster, 1999), hence increase the physical strength of the adsorbents (Zhang and Itoh, 2005). This has been confirmed in batch arsenic removal experiments, where no disintegration of the adsorbents happened after the treatment. It was also testified in the filtration experiment that the HIOFAA adsorbent was easily filtered with a clean filtrate with a filter paper (Grade 42, Whatman) which particle retention size was 2.5 µm. In contrast, a significant portion of particles passed through the similar filter paper during filtration of laboratory-made pure FeOOH suspension (Zeng, 2003). This observation indicated that no FeOOH suspension was disintegrated from the adsorbent.

Furthermore, silica on the surface of the particle could block or retard the transformation of initially formed FeOOH to crystalline iron oxides such as ferrihydrite, goethite and hematite. Arsenic adsorbent, the crystalline form of FeOOH , e.g. goethite and ferrihydrite, is not desired, since it has much less surface area than its amorphous form, and will reduce arsenic removal capability (Zhang and Itoh, 2005). In sum, the high arsenic removal capability of HIOFAA was attained in this system, and the high physical strength of adsorbent was also formed during the chemical process.

3.3. Effect of contact pH

The effect of solution pH on arsenic (V) removal by HIOFAA was studied in an initial pH range of 1.5 to 13.0. Fig. 5 shows that arsenic (V) was effectively removed in the initial pH range of 1.0 to 8.0, especially a removal peak around the pH of 2.5 appeared, with arsenic removal amount of as high as 23.0 g kg^{-1} . Therefore, HIOFAA was

Fig. 4. Nitrogen adsorption-desorption isotherm and pore size distribution of HIOFAA.

effective over a wider range of pH than the other adsorbents prepared by addition of extra iron source. For example, the adsorbents of IOLMS (Zhang and Itoh, 2005) which was synthesized by adding FeCl_3 solution on municipal solid waste incinerator melted slag, had the highest arsenic (V) removal at the pH of 1.5, but removal efficiency sharply decreased along with the increase of pH value.

The wide pH range for the utilization of HIOFAA could be attributed to aluminium hydroxide existed in the surface of the adsorbents, since it is effective in a wide pH range on arsenic removal (Mohan and Pittman, 2007). Accordingly, the removal mechanisms of HIOFAA for the arsenic include not only adsorption but also reaction with the oxide of both iron and aluminium on the surface of HIOFAA.

For comparing the removal of arsenic (V) by HIOFAA and the raw fly ash, test was conducted at pH 2.5 under the same conditions. The values of removal by the raw ash and HIOFAA were 11.2 g kg^{-1} and 23.0 g kg^{-1} , respectively, which indicated that the HIOFAA was much effective than the raw ash and its removal capability was 2 times of raw fly ash in this condition. This phenomenon could mainly attribute to the porous structure on the surface of HIOFAA and hereby the high surface area, e.g. the BET surface area for HIOFAA and RFA were 6.23 and $140.07 \text{ m}^2/\text{g}$, respectively. Loading of hydroxy compound on the surface of the residual fly ash increased the specific surface area for nearly 22 times compared to the raw ash, thus enhanced the efficiency of adsorption greatly.

3.4. Batch adsorption results

In the batch experiments, high arsenic concentrations were performed since wastewaters in refining plants (e.g., gold and silver refining plants) usually contain high arsenic concentrations. To obtain the adsorption equilibrium time, kinetic adsorption experiments were conducted, in which the HIOFAA dosage was 2 g l^{-1} and the initial arsenic concentration was 50 mg l^{-1} . Profile of contact time and the arsenic removal by the HIOFAA is presented in Fig. 6 (a), the results showed that arsenic removal increased sharply at the first 6 h and adsorption equilibrium was attained in 8 h.

The effect of adsorbent dose on arsenic (V) removal is showed in the Fig. 6 (b), the removal efficiency of arsenic (V) increased sharply with the increase of adsorbent dose at the first stage (the dose was below 20 g l^{-1}). However, the removal of arsenic increased slowly when more adsorbents were added into the solution till arsenic was fully adsorbed. In China the regulated limit on arsenic disposal in industrial wastewater is 0.05 mg l^{-1} . It is observed that 40 g of HIOFAA was sufficient to remove 50 mg Arsenic (V) from 1 l aqueous solution to meet the standard value.

Arsenic solutions at different initial concentration from 1.0 to 100 mg l^{-1} were treated with 2.0 g l^{-1} of adsorbent at pH 2.5. Fig. 6 (c)

Fig. 5. Effect of pH on arsenic adsorption by HIOFAA. Reaction conditions: arsenic concentration = 50 mg l^{-1} , adsorbent dose = 2 g l^{-1} , contact time = 15 h .

Fig. 6. Arsenic removal by HIOFAA as a function of (a) contact time, (b) HIOFAA dosage and (c) initial concentration. Reaction conditions for (a): arsenic concentration = 50 mg l^{-1} , adsorbent dose = 2 g l^{-1} , pH = 2.5; for (b): arsenic concentration = 50 mg l^{-1} , contact pH = 2.5, contact time = 8 h ; for (c): dosage = 2 g l^{-1} , contact pH = 2.5, contact time = 8 h .

shows that the effect of varying arsenic concentration against the removal efficiency of the arsenic. As expected, removal efficiency of arsenic decreased with the increasing of initial arsenic concentration, which manifested that the adsorption by the HIOFAA depended on the availability of binding sites for arsenic. When the initial arsenic concentration was 1.0 mg l^{-1} , the removal efficiency of arsenic could reach above 99% and the final concentration was decreased below 0.01 mg l^{-1} .

3.5. Arsenic adsorption isotherm model

In general, adsorption isotherm is used to describe the equilibrium state of adsorbate, adsorbent and solute at a given temperature. The

Table 2
Langmuir and Freundlich isotherm constants.

Langmuir ($\frac{C_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{C_e}{q_{\max}}$)			Freundlich ($\log q_e = \log K_F + \frac{1}{n} \log C_e$)		
$q_{\max}$ (mg g ⁻¹)	K_L (l mg ⁻¹)	R_L^2	n	K_F (mg g ⁻¹)	R_F^2
19.455	1.41	0.998	2.61	0.0717	0.876

C_e (mg l⁻¹): equilibrium arsenic concentration; q_e (mg g⁻¹): arsenic adsorption at equilibrium concentration; $q_{\max}$, K_L , n , K_F : adsorption constants.

arsenic adsorption data were analyzed with both Langmuir and Freundlich isotherm models since they are classical and simple to describe the equilibrium between metal ions onto adsorbent and metal ions in solution at a constant temperature. The respective empirical constants for the Langmuir and Freundlich isotherm models along with correlated coefficients (R^2) were presented in Table 2. The R^2 values obtained from the Langmuir and Freundlich isotherm were 0.998 and 0.876, respectively, indicating that the Langmuir isotherm fit better to HIOFAA than the Freundlich isotherm. Namely, the adsorption of arsenic (V) by HIOFAA was due to monolayer coverage of arsenic (V) onto homogeneous surface without interaction between adsorbed molecules. The Langmuir isotherm plot for the adsorption of arsenic (V) onto the HIOFAA was showed in Fig. 7, and the adsorption capacity ($q_{\max}$) obtained from the Langmuir isotherm model (Table 2) was 19.46 mg g⁻¹, which was much higher than that of the other adsorbents made from the ash by the way of adding iron source (e.g. 13.04 mg g⁻¹) (Fan et al., 2008), which should be ascribed to the high BET specific surface area of HIOFAA.

4. Conclusions

The study provided a potential application for the fly ash, as a kind of solid waste from thermal power stations, to be reused to control water pollution. An effective adsorbent for arsenic (V) removal from aqueous system has been successfully developed using high iron-containing fly ash as raw material. The adsorbent was in porous structure with specific surface area of 140 m²/g, which was 22 times of that of the raw ash. Moreover, the particle size of HIOFAA was 4 times of the original fly ash, thus it could be easily separated from aqueous solution after arsenic adsorption process. Batch experiments showed that HIOFAA was effective for arsenic (V) removal. About 40 g of HIOFAA was sufficient to remove 50 mg arsenic (V) from 1 l aqueous solution to meet the standard value for the industrial wastewater discharge.

The HIOFAA developed in this work has the following advantages over traditional iron-oxide adsorbents for arsenic removal: (1) avoiding

Fig. 7. Langmuir plot for the adsorption of arsenic onto the HIOFAA.

the addition of the extra iron source thus reducing the cost of arsenic treatment; (2) increasing the particle size and making it easy to separate from aqueous system after adsorption activity; (3) changing the surface structure of powder particle into porous structure, thus increasing the surface area and promoting arsenic removal capacity; (4) easy for preparation since all the chemical reactions occur under mild conditions.

Acknowledgements

This work was financially supported by the National Basic Research Program of China (2007CB407303) and the National Key Technology R & D Program (2008BAC32B03).

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